



Microwave-induced synthesis of electrical conducting gum acacia-graft-polyaniline

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ABSTRACT

In the presence of catalytic amount of ammonium peroxydisulfate (APS) as oxidant/initiator, the grafting of polyaniline (PANI) on to gum acacia (GA) was done under mild acidic condition using microwave irradiation. The synthesis could be accomplished rapidly under atmospheric conditions. The grafting condition was optimized by varying the microwave power, exposure time and concentrations of initiator, acid, monomer and gum acacia. The copolymer was characterized using UV/vis and Fourier-transform infrared (FTIR) spectroscopy, ¹H NMR, thermogravimetric analysis (TGA) and transmission electron microscopy (SEM). The gum acacia-graft-polyaniline (GA-g-PANI) exhibited pH switching electrical conducting behavior dependent on the grafting extent and on the pH of the material. The findings have been discussed and a plausible mechanism for graft copolymerization has been proposed.

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1. Introduction

The modification of carbohydrate polymers with conducting polymers may lead to the preparation of multifunctional electrical conducting biomaterials. It will be interesting in terms of compatibility with environment and biological systems. Gum acacia (GA) is a complex arabinogalactan which contains a small proportion of proteinaceous materials and has been classified as arabinogalactan–protein complex (Islam, Phillips, Sijivo, Snowden, & Williams, 1997). The gum has a highly variable composition and physical and chemical properties can vary considerably depending upon the source of the nodules (Islam et al., 1997). It is composed of D-galactose, L-arabinose, L-rhamnose, D-glucuronic acid and 4-O-methyl-D-glucuronic acid (Sanchez, Renard, Robert, Schmitt, & Lefebvre, 2002). It has been reported that inorganic salt complexes of gum acacia can behave as a superionic electrical conductor (Malik, Gupta, & Sarkar, 2002). In general, incorporation of conducting polymers such as polyaniline (PANI) into a flexible matrix of GA should result in good processability along with the electrical conductivity, chemical stability toward dopants and thermal stability.

Copolymers of PANI with biopolymers have also been conventionally synthesized using various initiators such as polyaniline giving a semi interpenetrating network (IPN) based on chitosan (Shin et al., 2005), gum arabic-graft-polyaniline (Tiwari, 2007) and polyaniline-cellulose acetate (Ahmed, Mohammada, & Rahman, 2004). The conducting PANI grafted biopolymers are interesting candidates for the development of biopolymer based electronic

materials for the environmental favorable technologies but their synthesis involve quite exhaustive and time consuming procedures.

In chemical processing, microwave irradiation is emerging as efficient tool for the chemical processing (Galema, 1997) and has been exploited in various fields of chemistry including polymers. Main advantage is that it results in almost instantaneous ‘in core’ processing of materials in a homogeneous and selective manner (Singh, Premlata, Tiwari, & Sharma, 2007). Under microwave irradiation, homopolymerization of polyacrylamide (Singh, Tiwari, Kumari, & Sharma, 2007) and graft copolymerization of polyacrylamide, polyacrylonitrile onto guar gum (Singh, Tiwari, Tripathi, & Sanghi, 2004; Singh, Tripathi, Tiwari, & Sanghi, 2004), chitosan (Singh, Tiwari, Tripathi, & Sanghi, 2006; Singh, Tripathi, et al. 2004) and potato starch (Singh, Tiwari, Pandey, & Singh, 2006; Singh, Tiwari, Pandey, & Singh, 2007) have been reported without radical initiators or with very low initiator concentration. The present communication for the first time reports on the peroxydisulfate initiated synthesis of electrical conducting biopolymer based on GA and PANI under microwave conditions. In the study, optimization of grafting conditions, reaction mechanism, morphology, conducting, and thermal behavior of the grafted material were investigated.

2. Experimental

GA (E. Merck, M_w 1.64×10^5) was used after purification (Sanchez et al., 2002). Aniline (E. Merck) was distilled prior to use and ammonium peroxydisulfate (Aldrich) was used without further purification. The deionized water was used for all the reactions.

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A Milestone (Model no. PN 44072, 1600W) laboratory microwave oven was used for all the experiments. Ocean optics HR 4000 high-resolution spectrophotometer was used for recording UV/vis spectra of aqueous solution of samples. FTIR spectra were recorded on PK-1310 Perkin–Elmer instrument by making pellet with dehydrated KBr in reflectance mode. ^1H NMR was done on Bruker DRX-300 NMR spectrometer with $(\text{CD}_3)_2\text{SO}$ as solvent and SiMe_4 as internal standard. TGA was recorded on Rigaku Thermoflex PTC-10A with 10°C per min heating rate and aluminium as the reference material. SEM was performed on a JEOL-840 by making a film of desirable size and coated with gold. For the conductivity measurements, Kithley four-probe system with 224 programmable current source, 181 nano-voltmeter and 195A digital multimeter were used.

The percentage and efficiency of grafting were calculated by the following equations (Singh, Tiwari, Tripathi, & Sanghi, 2005)

$$\% \text{ Grafting } (\%G) = \frac{W_1 - W_2}{W_0} \times 100 \quad (1)$$

$$\% \text{ Efficiency } (\%E) = \frac{W_1 - W_2}{W_2} \times 100 \quad (2)$$

where W_1 , W_0 and W_2 denote, the weights of GA-g-PANI, GA and the aniline monomer, respectively.

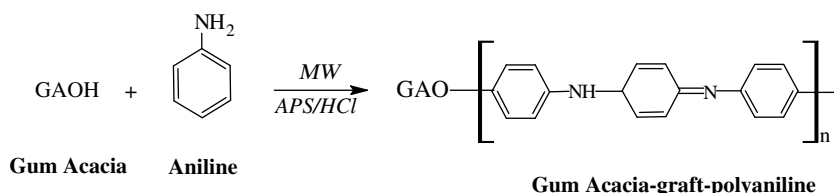
2.1. Graft co-polymerization

2.1.1. Conventional synthesis of GA-g-PANI

In a 150 ml flask, GA (8 g/L), aqueous HCl (1.25 M) and aniline (0.2 M) were dissolved in a 25 ml of deionized water and the reaction mixture was thermostated at $40 \pm 0.2^\circ\text{C}$ with continuous stirring. After 30 min, 0.5 M ammonium peroxydisulfate (APS) was added and this was taken as zero time. Grafting was allowed for 2 h. The reaction mixture was first neutralized with 5% aqueous NaOH, after that the copolymer was precipitated with absolute ethanol. The resulting precipitate was washed with *N*-methylpyrrolidone (NMP) in order to separate PANI (homopolymer) from the copolymer (Scheme 1). The copolymer was ground to a fine powder and low molecular weight PANI oligomers were removed from GA-g-PANI by soxhlation with acetone. Finally, the products were dried for several days in a vacuum oven at 50°C (%G-149, %E-64.09).

2.1.2. Microwave-induced synthesis of GA-g-PANI

GA (8.0 g/L), hydrochloric acid (2.0×10^{-3} M) and aniline (0.1 M) were dissolved in 25 ml of water. To this solution, $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (40×10^{-5} M) was added with continuous stirring. The homogeneous reaction mixture was then irradiated under the laboratory microwave oven at definite microwave power and exposure time. After completion of the reaction, the mixture was neutralized with 0.5% aqueous NaOH and the copolymer was precipitated with absolute ethanol. The resulting precipitate was washed with NMP and the copolymer was ground to a fine powder and low molecular weight PANI oligomers were removed by soxhlation with acetone. Finally, the products were dried in a vacuum oven.



Scheme 1. Synthesis of gum acacia-graft-polyaniline via microwave-accelerated oxidative radical polymerization.

2.2. Conductivity measurement

The four-point probe method was used for the determination of electrical conductivity of the GA-graft-PANI by making rectangular pressed pellets (pressure 5 tons/cm²).

According to the four-point probe methods (Zhang, 2007) the resistivity can be calculated using the relation:

$$\rho = 2\pi S(V/I) \quad (3)$$

where S is the probe spacing (mm), which was kept constant, I is the supplied current in mA, and the corresponding voltage was measured in mV.

Conductivity can be computed using the relationship:

$$\sigma = 1/\rho \quad (4)$$

3. Results and discussions

3.1. Optimization of grafting conditions

The optimum grafting condition was determined via varying the microwave power, exposure time and concentrations of APS, HCl, aniline and GA, while total volume of the reaction mixture was fixed at 25 ml. It was observed that under microwave irradiation very low concentration of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and hydrochloric acid was required for the graft copolymerization of PANI and GA. The maximum %E achieved was 98.30%.

3.1.1. Effect of microwave power

The grafting was carried out by giving 30 s exposure to the reaction mixture at different microwave powers ranging from 200 to 600 W using [APS] 5×10^{-5} ; [HCl] 1×10^{-3} ; [aniline] 0.05 and [GA] 2.0 g/L. %G and %E both increases initially with the increasing microwave power up to 500 W, this may be due to more availability of microwave energy at high microwave power, causing generation of more monomer and macro radicals that resulted in high % grafting (Fig. 1). After 500 W, decrease in %G and %E both may be attributed to the formation of more homopolymer at high microwave power or to some decomposition of the graft copolymer.

3.1.2. Effect of exposure time

The effect of exposure time was monitored by irradiating the reaction mixture for various time periods ranging from 20 to 60 s at 500 W microwave power keeping the other variables fixed; [APS] 5×10^{-5} ; [HCl] 1×10^{-3} ; [aniline] 0.05 and [GA] 2.0 g/L. The %G and %E both increased rapidly up to 40 s. exposure and then decreased (Fig. 2). This could be attributed to the availability of extra microwave energy at higher exposure time however, on prolonged exposure (after 40 s), %G decreases probably due to some decomposition of the graft copolymer.

3.1.3. Effect of APS concentration

The %G increased with an increase in initiator/oxidant concentration, and was optimum at 40×10^{-5} M of APS at fixed concentrations of HCl (1×10^{-3}); aniline (0.05 M) and GA (2.0 g/L) at

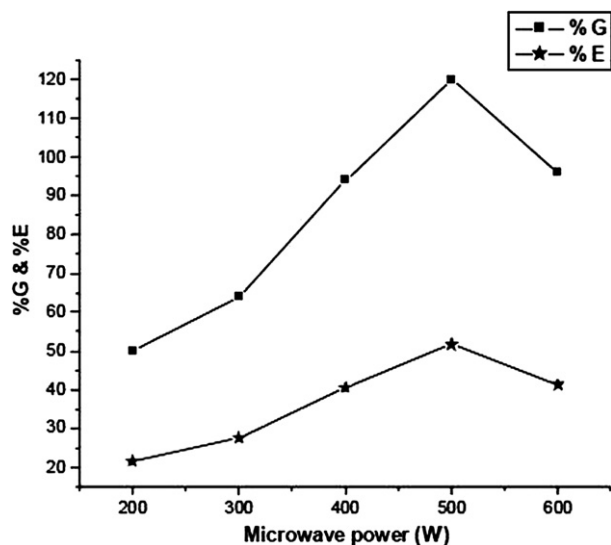


Fig. 1. Effect of microwave power keeping other variables constant at [APS] 5×10^{-5} ; [HCl] 1×10^{-3} ; [aniline] 0.05 and [GA] 2.0 g/L and exposure time 30 s.

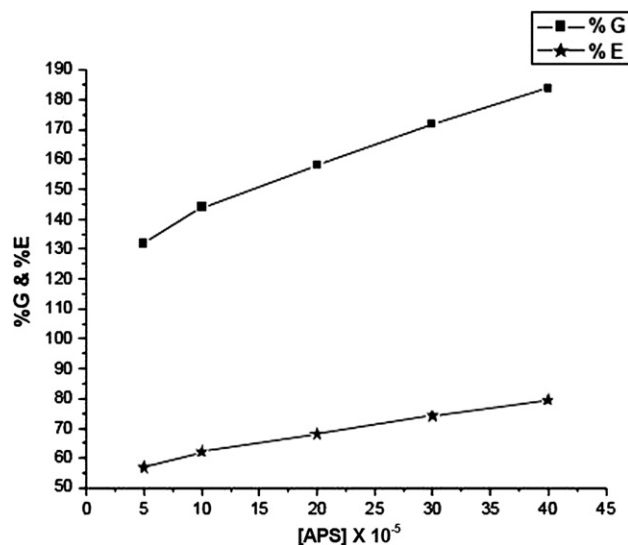


Fig. 3. Effect of APS concentration at fixed [HCl] 1×10^{-3} ; [aniline] 0.05 and [GA] 2.0 g/L at 500 W microwave power and exposure time 40 s.

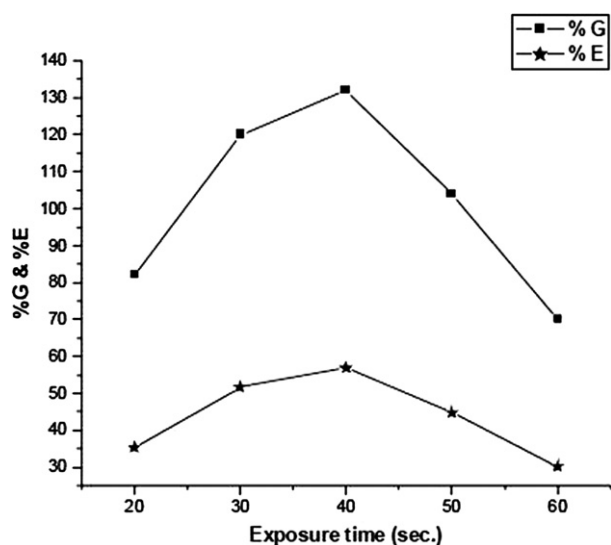


Fig. 2. Effect of exposure time keeping other variables constant at [APS] 5×10^{-5} ; [HCl] 1×10^{-3} ; [aniline] 0.05 and [GA] 2.0 g/L and microwave power 500 W.

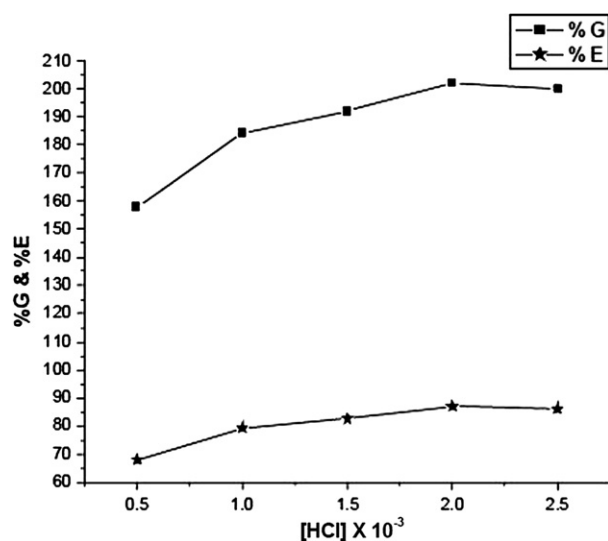


Fig. 4. Effect of HCl concentration at fixed concentration of 40×10^{-5} M APS, 0.0–5 M aniline and 2.0 g/L GA at 500 W microwave power and 40 s exposure.

500 W microwave power and 40 s exposure (Fig. 3). It may be due to the fact that at this concentration range, the produced sulfate ion radical moieties activate the backbone of GA and simultaneously produce the PANI ion radical, followed by this grafting of the monomers takes place onto the GA backbone. Therefore, %G and %E both increased with an increase in the concentration of peroxydisulfate.

3.1.4. Effect of HCl concentration

The effect of acid concentration was also studied in the range $0.5\text{--}2.5 \times 10^{-3}$ M at a fixed concentration of APS (40×10^{-5} M), aniline (0.05 M) and GA (2.0 g/L) at 500 W microwave power and 40 s. exposure. It was observed that both %G and %E increase with an increase in the acid concentration (Fig. 4) up to a certain limit after that it become constant. The finding may be due to more protonation of the aniline monomer which accelerated the generation of PANI ion radicals.

3.1.5. Effect of aniline concentration

The increase in the concentration of monomer from 0.05 to 0.25 M resulted in an increase of %E (up to 0.1 M), under the fixed concentration of APS (40×10^{-5} M), HCl (2×10^{-3} M) and GA (2.0 g/L) at 500 W microwave power and 40 s. exposure (Fig. 5). The increase in %G and %E may be due to the formation of more PANI radical ions, while generating more grafting sites and availability of extra aniline monomers for grafting. With an increase in the concentration beyond 0.1 M, %E decreases slightly, which is probably due to more formation of PANI homopolymer.

3.1.6. Effect of GA concentration

The effect of GA concentration was studied in the range of 2–10 g/L with fixed concentration of APS (40×10^{-5}), HCl (2×10^{-3}) and aniline (0.2 M) at 500 W microwave power and 40 s. exposure time (Fig. 6). It was found that the %E increased up to 8 g/L GA concentration, it is due to more availability of macro

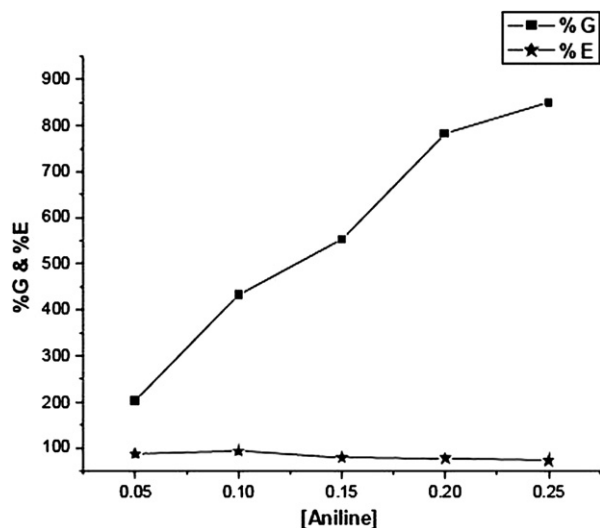


Fig. 5. Effect of aniline concentration under the fixed concentration of 40×10^{-5} M APS, 2×10^{-3} M HCl and 2.0 g/L GA at 500 W microwave power and 40 s exposure.

radicals for the grafting. Beyond it %E decreased, which may be due to the increase in the viscosity of the reaction medium causing hindrance to the reaction.

3.2. Mechanism for graft copolymerization

The large number of —OH groups located at GA macromolecule behave as if they are anchored to an immobile raft (GA macromolecule) and their localized rotation (Galema, 1997) therefore will be observed under microwaves, resulting in dielectric heating which cause bond breakage and thus generating radical sites. Furthermore, microwaves also have a specific effect in lowering of the energy of activation of the reactions and these two facts accelerate the rapid graft copolymerization. In the light of the above a plausible oxidative-free radical mechanism for the PANI grafting on to GA under microwave irradiation has been proposed. As previously reported (Singh, Tiwari, & Sanghi, 2005; Singh, Tiwari, Singh, Shukla, & Sanghi, 2006), where we have explored free radical copolymerization of vinyl monomers with carbohydrate polymers using peroxydisulfate, a chain mechanism is involved due to formation of sulfate ion radicals ($\text{SO}_4^{\cdot-}$), which are well known ion chain carriers for the graft copolymerization (Scheme 2). At

the same time peroxydisulfate stimulates the oxidative polymerization reaction of aniline (Ding, Padias, & Hall, 1999) via a medium of cationic radicals and form PANI and PANI radicals (Scheme 3). Finally GA macro radicals and PANI cation radicals are combined to form GA-g-PANI graft copolymer.

$\text{SO}_4^{\cdot-}$ is the primary radicals, generated from the APS by the reduction of one electron, expressed as R^{\cdot} in the Scheme 4. Simultaneously, APS generate SO_4^{2-} ions by the reduction of two electrons and act as oxidant. They initiate the oxidative polymerization of aniline, as the polymerization of monomer is reported to be faster than the H abstraction from the biopolymer backbone (Singh et al., 2007). The macro radicals GAO^{\cdot} may be generated by the abstraction of H by the growing PANI ion radical (PANI^{\cdot}) in the acid medium, which may add onto the GAO^{\cdot} macro radicals generating new radical GAO-PANI^{\cdot} and these chains will grow and combined with other GAO-PANI^{\cdot} chains to give a graft copolymer (Scheme 4).

3.3. Characterization of GA-g-PANI

A representative GA-g-PANI sample (98.30 %E) was characterized by UV/vis, FTIR, ^1H NMR, TGA and SEM analysis.

3.3.1. UV/vis Spectra

The UV/vis spectra of GA-g-PANI at pH 1 showed a broad absorption band at 300–465 nm due to overlapping of arabinogalactan components of GA, $\pi-\pi^*$ transition of benzenoid rings and polaronic peak of grafted PANI. The peak at 800 nm was assigned bipolaronic transitions of grafted PANI, while GA-g-PANI at pH 12 showed a peak at 620 nm due to the $\pi-\pi^*$ transition of quinoid rings of the grafted PANI. These curves evidenced that the grafted PANI showed interconversion of emeraldine salt to emeraldine base (protonation–deprotonation or doping–dedoping) transition and bears emeraldine oxidation state at different pH (Ghosh, Chakrabarti, & Siddhanta, 1999; Sengupta, Barik, & Adhikari, 2006). Where as, GA showed a broad absorption band at 298 nm due to presence of arabinogalactan components in the GA (Sanchez et al., 2002). Furthermore, the characteristic peaks of arabinogalactan components and PANI both were observed in the graft copolymer and it confirmed the grafting of PANI on to GA backbone. UV/vis spectra suggested the chemical structure, conjugation, electronic transition and doping–dedoping behavior of GA-g-PANI (Fig. 7).

3.3.2. FTIR Spectra

FTIR spectra of GA-g-PANI showed characteristic peaks of PANI as well as GA, it had peaks at ν_{max} 3040–3266 cm^{-1} (N–H stretching of hydrogen bonded amino groups and free O–H stretching vibration); ν_{max} 3024 cm^{-1} (aromatic C–H stretching), ν_{max} 2930 cm^{-1} and ν_{max} 2864 cm^{-1} (aliphatic C–H stretching), ν_{max} 1632 cm^{-1} (C=O stretching of carbonyl group, typical saccharide absorption), ν_{max} 1526 cm^{-1} (C=C stretching of quinoid rings), ν_{max} 1423 cm^{-1} (C=O stretching of —COOH group and —OH bending of acid group, respectively), ν_{max} 1462 cm^{-1} (C=C stretching vibration of benzenoid rings) and at ν_{max} 1284 cm^{-1} (C–N stretching). The peak corresponding to the N=O=N bending vibration of PANI shifted to lower wave number (ν_{max} 1110 cm^{-1} from ν_{max} 1130 cm^{-1}). The shift can be attributed to the hydrogen bonding between gum acacia and imine group of the grafted PANI chains. The other observed bands were at ν_{max} 1030 cm^{-1} (O–H bending), at ν_{max} 1076 cm^{-1} (characteristic peaks of a saccharide structure; C–O stretching) and at ν_{max} 830 cm^{-1} (aromatic C–H bending vibration band due to the 1,4-disubstituted benzene ring). While FTIR spectra of the GA showed typical polysaccharide absorption; ν_{max} 3418 cm^{-1} (stretching of —OH group), ν_{max} 2932 cm^{-1} (C–H stretching), ν_{max} 1634 cm^{-1} (C=O stretching of carbonyl group, typical to a saccharide absorption), ν_{max} 1720 cm^{-1} and ν_{max}

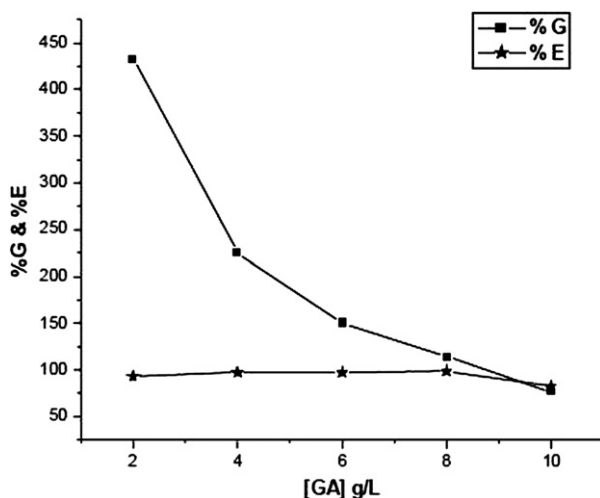
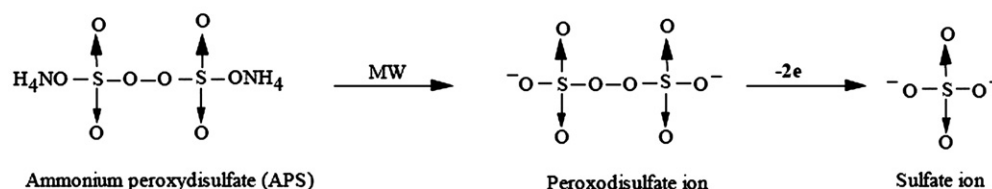
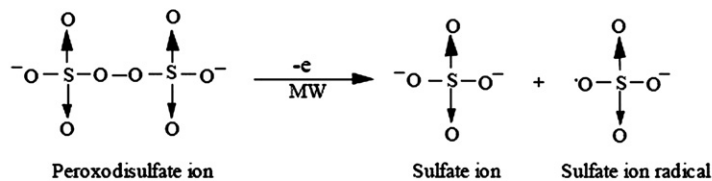


Fig. 6. Effect of GA concentration at constant [APS] 40×10^{-5} , [HCl] 2×10^{-3} and [aniline] 0.1 at 500 W microwave power and 40 s exposure.

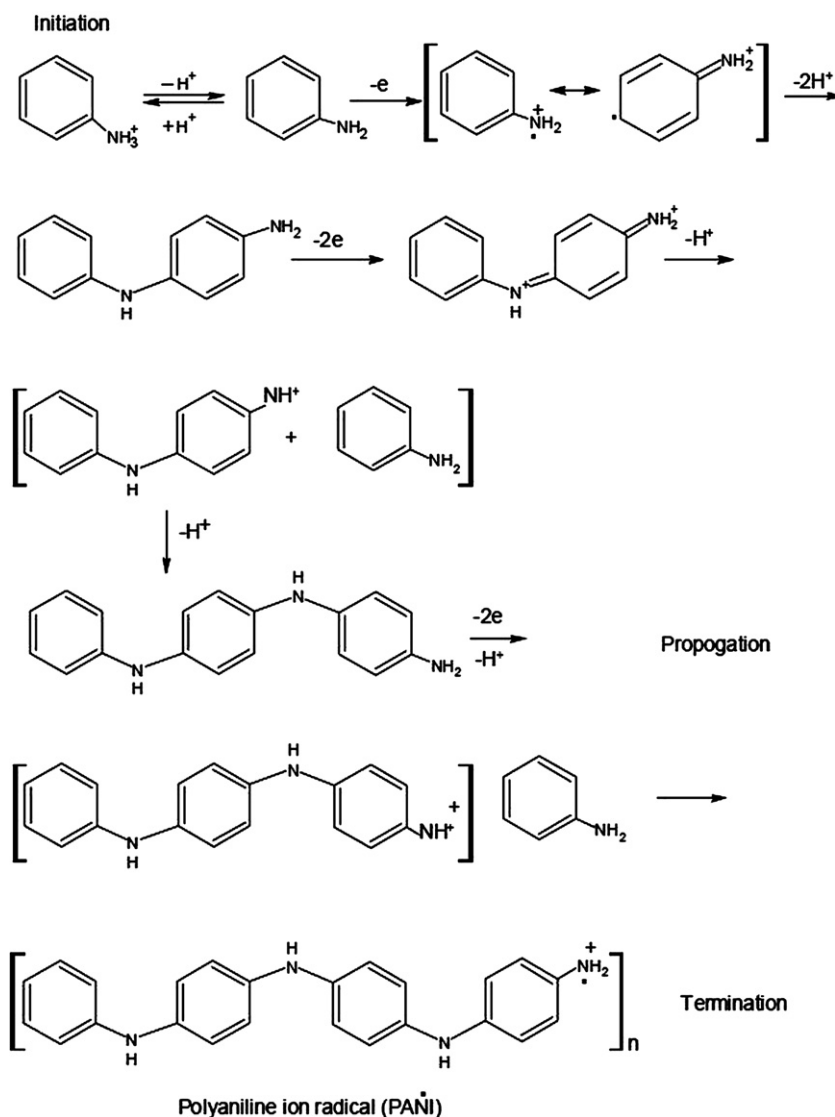
1) Oxidizing action of APS



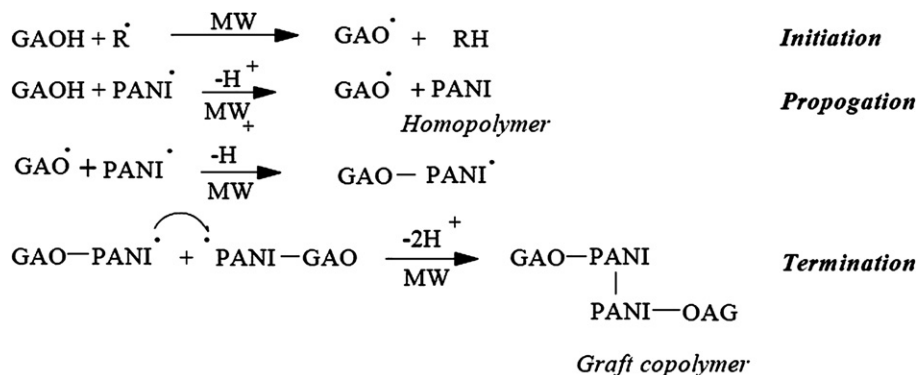
2) APS act as initiator



Scheme 2. Generation of primary radicals under microwave irradiation.



Scheme 3. Formation of secondary radicals under microwave irradiation.



(Where GAOH represents gum acacia, R is primary radicals generated from represents PANI cation radicals Scheme-2 and PANI[•] i.e. generated from Scheme-3)

Scheme 4. Microwave-accelerated graft copolymerization of PANI on to GA.

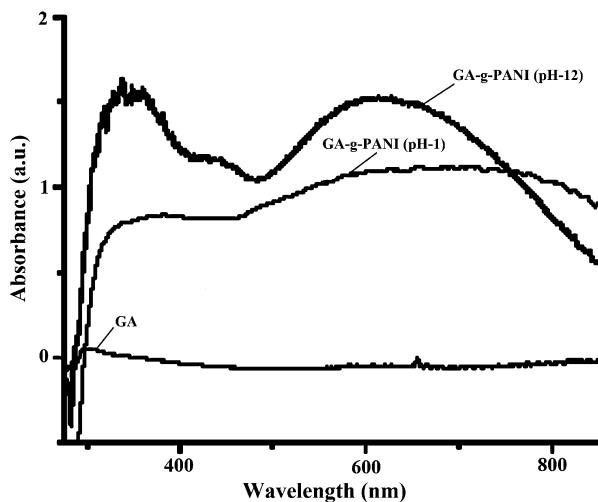


Fig. 7. UV-vis spectra of GA-g-PANI at pH 1; GA-g-PANI at pH 12 and GA.

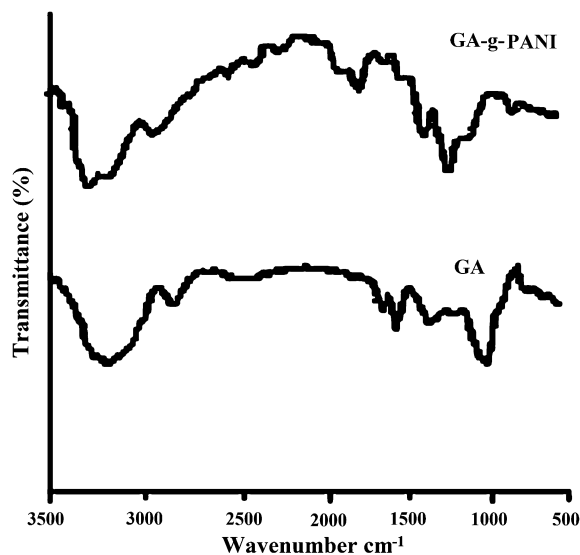


Fig. 8. FTIR spectra of GA-g-PANI and GA.

1423 cm⁻¹ (due to C=O stretching of —COOH group and —OH bending of acid group, respectively) and another characteristic peak of carboxylic group found at ν_{max} 1254 cm⁻¹ due to C—O stretching. Hence, FTIR studies further supported the grafting of PANI on to GA (Fig. 8).

3.3.3. ¹H NMR

¹H NMR of GA had a peak at δ 2.51 (acid protons present in the sugar moiety) and δ 3.48 ppm (sugar protons) while GA-g-PANI showed peaks at δ 2.50, δ 3.50, δ 4.55, 7.17, 7.34 and 7.51 ppm which corresponded to acid protons, sugar protons, —NH— and protons of benzene ring, respectively. The ¹H NMR studied confirmed the graft copolymerization of PANI on to GA (Fig. 9).

3.3.4. TGA spectra

The thermal resistance of GA-g-PANI was determined by TGA taking GA as reference. TGA indicated that the decomposition of the graft copolymer onsets at 210 °C and 57% weight loss up to 600 °C, while decomposition in the gum acacia onsets at 175 °C with 88% weight loss up to 600 °C. It was observed that the weight loss was slower in the case of the copolymer indicating it to be more thermally stable than the GA (Fig. 10).

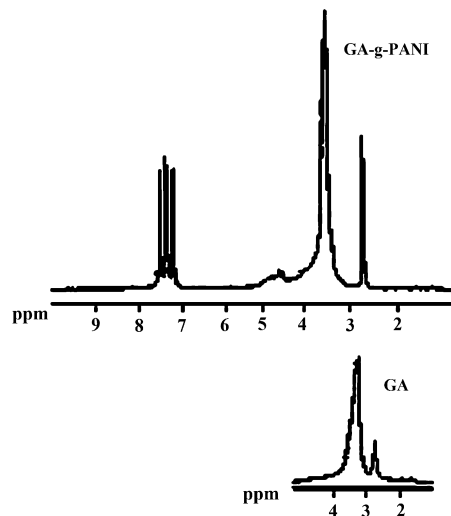


Fig. 9. ¹H NMR spectra of GA-g-PANI and GA.

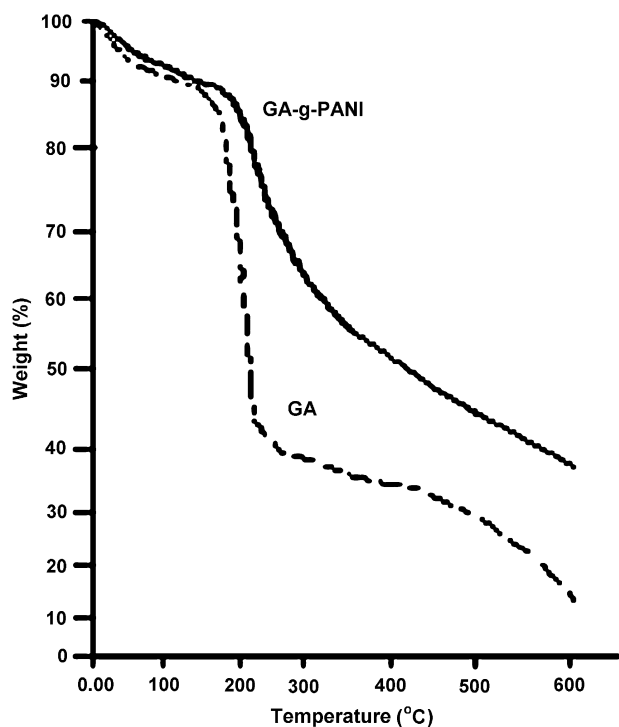


Fig. 10. TGA spectra of GA-g-PANI and GA.

3.3.5. SEM

The surface topography of the GA-g-PANI was studied by SEM and compared with the GA. The exterior surface of the GA-g-PANI appears to be porous and fluffy and quite different from that of the parent GA, which has irregular rocky surface. Hence, the surface evidence supports the grafting of PANI on to the GA (Fig. 11).

3.4. Electrical conductivity

The electrical conductivity of microwave synthesized graft copolymer (material with optimum %E) was measured with reference to conventionally synthesized GA-g-PANI, PANI and GA. The results are summarized in Table 1. The value of electrical conductivity of GA improved for about 10^4 times after grafting of PANI; moreover, it was observed that the conductivity of the microwave synthesized copolymer was almost double to that of the conventionally synthesized copolymer. This higher conductivity is due more incorporation of PANI grafts under microwave conditions.

Table 1

Electrical conductivity of PANI, GA, conventionally synthesized GA-g-PANI (CS) and GA-g-PANI (M_W) synthesized under microwave irradiation at 35 °C

Sl. No.	Materials	pH	%E	Electrical conductivity ($S\text{ cm}^{-1}$)
1.	PANI	1	–	0.742
2.	GA	1	–	4.73×10^{-6}
3.	GA-g-PANI (CS)	1	64.09	5.18×10^{-2}
4.	GA-g-PANI (M_W)	1	98.30	9.72×10^{-2}

Table 2

Effect of % grafting on the electrical conductivity of GA-g-PANI (M_W) at 35 °C

Sl. No.	Materials	pH	%G	Electrical conductivity ($S\text{ cm}^{-1}$)
1.	GA-g-PANI	1	50	7.45×10^{-5}
2.	GA-g-PANI	1	120	6.85×10^{-4}
3.	GA-g-PANI	1	184	5.08×10^{-3}
4.	GA-g-PANI	1	432	1.97×10^{-2}
5.	GA-g-PANI	1	850	9.72×10^{-2}

Table 3

Effect of pH on the electrical conductivity of GA-g-PANI (M_W) at 35 °C

Sl. No.	Materials	pH	%E	Electrical conductivity ($S\text{ cm}^{-1}$)
1.	GA-g-PANI	1	98.30	9.72×10^{-2}
2.	GA-g-PANI	3	98.30	2.98×10^{-2}
3.	GA-g-PANI	7	98.30	8.41×10^{-3}
4.	GA-g-PANI	12	98.30	4.55×10^{-6}

The effects of % grafting and pH on the electrical conductivity of the material are summarized in the Tables 2 and 3. It was observed that with increase in the % grafting, the electrical conductivity of the GA-g-PANI continuously increased, indicating that the grafting of PANI is responsible for the electronic conduction in GA-g-PANI. The conductivity of GA-g-PANI increased on exposure to HCl vapor and decreased when exposed to NH_3 vapors due to conversion of emeraldine salt to emeraldine base (Chabukswar, Pethkar, & Athawale, 2001; Gospodinova & Terlemezyan, 1998). Therefore due to its redox surface and pH switching behavior, the biomaterial may find use in sensor applications.

4. Conclusions

A conducting biomaterial based on GA and PANI was synthesized under microwave irradiation using catalytic amount of peroxydisulfate. The synthesis was performed under acidic medium within few

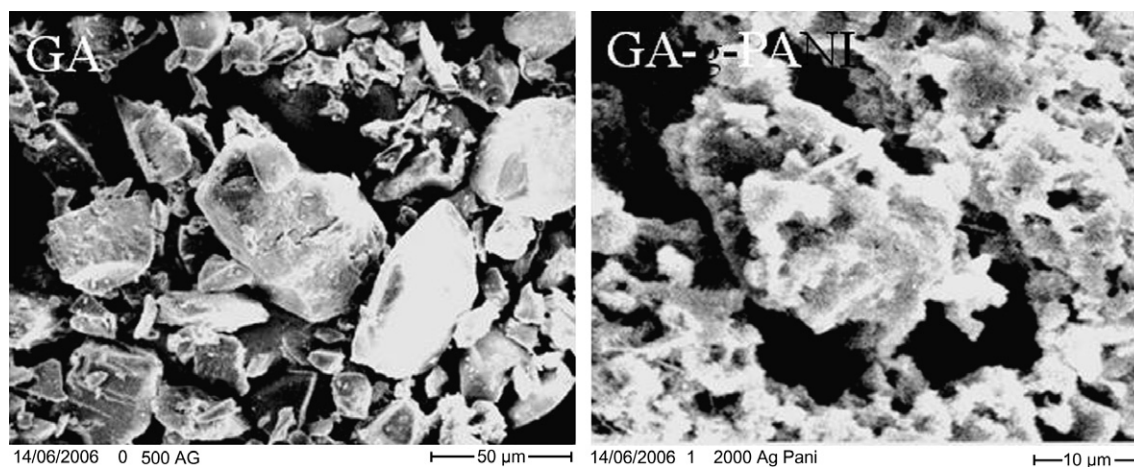


Fig. 11. SEM pictures of GA-g-PANI and GA.

seconds. The optimum grafting was observed when the reaction mixture containing 40×10^{-5} M $(\text{NH}_4)_2\text{S}_2\text{O}_8$; 0.1 M aniline; 2×10^{-3} M hydrochloric acid and 8.0 g/L GA was exposed to 500 W microwave power for 40 s. The grafted gum showed pH switching conducting behavior that was dependent on the grafting extent. The observed controlled multifunctional electronic properties of the material may be usefully exploited for technological applications.

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